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(71) Applicant: UNILEVER PLC, Unilever House Blackfriars P O Box 68, London EC4P 4BQ (GB)
(84) Designated Contracting States: GB

(71) Applicant: UNILEVER NV, Burgemeester s'Jacobplein 1 P.O. Box 760, NL-3000 DK Rotterdam (NL)
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(72) Inventor: Finch, Timothy David, 60 Sunningdale Drive, Bromborough Wirral Merseyside (GB)
Inventor: Wilde, Raymond John, 22 Vanderbyl Avenue, Spital Wirral Merseyside (GB)

(74) Representative: Mole, Peter Geoffrey et al, UNILEVER PLC Patents Division P.O. Box 68 Unilever House, London EC4P 4BQ (GB)

(54) **Process for manufacturing detergent powder.**

(57) Photobleach, such as zinc or aluminium phthalocyanine sulphonate is incorporated into detergent powder by spraying in solution or suspension. Reduced loss of photobleach during processing and during storage of the resultant powder is achieved by adopting the process.

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PROCESS FOR MANUFACTURING DETERGENT POWDER

This invention relates to a process for manufacturing detergent powder containing a photobleach.

Photobleaches are now being introduced into washing powders, particularly into those sold in the sunnier countries of Europe and in South America. The most convenient way of incorporating photobleaches such as zinc or aluminium phthalocyanine sulphonate into detergent powders prepared by spray-drying would be to incorporate them into the aqueous crutcher slurry, together with other components such as detergent active compound and builders. However, when this method is used in practice, loss of photobleach occurs, and the resulting powder has poor stability during storage.

According to this invention there is provided a process for the manufacture of a particulate detergent fabric washing product which comprises forming a spray-dried or granulated base powder and combining it with a photobleach compound, characterised in that the combination is effected by spraying a solution or suspension of the photobleach onto a powder comprising the base powder.

European patent application No 0 057 088 (Procter & Gamble Limited) discloses spraying an aqueous solution of a photobleach and of an amorphous phosphate onto a mixture of a storage-sensitive detergent and a hydratable salt. In contrast to the disclosure, the present invention provides a generally applicable process for incorporation of a photobleach into spray-dried or granulated powders without loss during processing or subsequent storage.

One of the key properties of photobleaches is their ability to sorb onto the fibres of an article. This property is extremely concentration dependent and is also sensitive to modifications in molecular structure, which are believed to occur when photobleaches are incorporated into a crutcher slurry. In order to make good what would amount to a loss of the original photobleach, were it decided to process powder by including photobleach in the slurry, it would be necessary to increase the initial concentration to obtain the desired performance in the spray-dried product. That would be an extremely expensive measure. Even that would not counteract the loss which occurs during storage when photobleach is incorporated into a powder by a slurry-making and spray-drying route.

It is preferred that the photobleach is a zinc or aluminium phthalocyanine sulphonate and that it is sprayed onto the spray-dried or granulated base powder in aqueous solution or suspension. While the solution or suspension may be of any desired concentration, better control of the amount of photobleach incorporated into the powder is achieved if it is very dilute, for example 0.001 to 0.2% by weight.

The solution or suspension may be sprayed onto the spray-dried or granulated powder in any desired fashion, for example in an inclined pan granulator such as an Eirich

pan (trade mark), in a fluidised bed or rotating drum mixer, or onto the powder on a moving belt, or in a curtain as it falls from one level in the plant to another.

The solution or suspension preferably contains an inorganic acid salt such as sodium dihydrogen phosphate, sodium bicarbonate and sodium bisulphate, which has been discovered to improve the physical properties of the sprayed powder.

The question of brightness or apparent whiteness is one which really only applies to fabric washing, other indices being appropriate in the dishwashing field for example. Consequently, this invention is only applicable to fabric washing products. However, as implied above it is applicable to both spray-dried products prepared by hot-spray-drying of an aqueous crutcher slurry, or to granulated products prepared by absorbing the liquid components of the composition onto the solid ones to provide a balanced, free-flowing particulate solid.

The major components of the fabric washing product in accordance with the invention are one or more anionic and/or nonionic surfactants and one or more detergency builders, together with a number of optional components.

Typical anionic detergent active compounds, which may be present in amounts of from about 2 to 35% by weight of the finished compositions are sodium alkylbenzene sulphonates, preferably the C_{10}-C_{16} alkyl compounds, sodium primary and secondary alkyl sulphates, preferably the C_{10}-C_{22} alkyl sulphates, sodium olefine sulphonates, preferably the C_{10}-C_{18} sulphonates and sodium alkane sulphonates. Soaps of fatty acids may also be present, preferably the sodium and potassium salts of C_{10}-C_{22} fatty acids, both saturated and unsaturated.

Where soap is the sole anionic surfactant it may be present in an amount up to about 65% by weight of the finished composition, down to about 0.2% by weight when other anionic surfactants are present. Typical soaps which can be used are those formed from coconut oil, tallow, hydrogenated tallow, hydrogenated rapeseed oil and natural oils containing high proportions of oleic acid such as sunflower oil.

Typical nonionic surfactants are ethoxylated primary and secondary alcohols of from 8 to 25 carbon atoms containing from 3 to 25 moles of ethylene oxide per mole of alcohol. These materials may generally be present in an amount of from 1 to 25% by weight, based on the weight of the finished composition.

Typical detergency builders which can be used are the water-soluble phosphates, carbonates and aluminosilicates, particularly the sodium and potassium salts of these compounds. Organic builders may also be used, examples being sodium carboxymethyloxysuccinate, sodium citrate, sodium polyacrylates and sodium nitrilotriacetate. Any of these compounds, or any other builder compound, in any suitable mixture, may be used in amounts of from 5 to 60% by weight of the finished composition. Other components which will normally be present are bleaching agents and precursors therefore, such as tetraacetylethylene diamine, corrosion inhibitors, anti-redeposition agents, fluorescers, stabilisers, enzymes and substantial proportions of water.

The process of the invention will be illustrated in the following examples, Example 1 being a comparative example.

Example 1

Aluminium phthalocyanine sulphonate (AIPCS) was incorporated into a crutcher slurry having the composition quoted below in the amounts shown in Table 1. The slurry was then spray-dried to a powder and the content of AIPCS was determined by visible absorption spectroscopy.

	% by weight
Sodium dodecylbenzene sulphonate	16.8
Sodium tripolyphosphate	17.5
Sodium silicate (ratio 2:1)	8.1
Sodium carboxymethyl cellulose	0.6
Sodium sulphate	17.0
AIPCS	See Table 1
Water and minor components	40.0
	100.0

Table 1

AIPCS incorporated in slurry (%)	AIPCS content by analysis	% loss
0.0087	0.007	19.6
0.0100	0.008	20.0
0.0200	0.016	20.0

Example 2

A carefully measured quantity of AIPCS solution having a concentration of 0.299% was sprayed onto spray-dried base powder from an Amicon (trade mark) pressure vessel fitted with a Delavan Watson (trade mark) single fluid spray jet at an operating pressure of 70 psig. The base powder (20 Kg) was agitated by being rotated in a 1 metre pan granulator inclined at an angle of 50-54° to the horizontal and rotating at a speed of 22 rpm.

The formulation of the base powder was

	% by weight
Sodium dodecylbenzene sulphonate	25.0
Sodium tripolyphosphate	26.0
Sodium silicate	12.0
Sodium carboxymethyl cellulose	1.0
Sodium sulphate	25.0
Water and minor components	11.0
	100.0

The content of AIPCS was determined as before and was found to be the same as the theoretical value, within the experimental error.

Example 3

An aqueous solution containing AIPCS (0.08%) and sodium dihydrogen phosphate (25%) was sprayed from an Amicon (trade mark) pressure vessel onto a falling curtain of a spray-dried base powder having the composition shown in Example 2. The rate of flow of the powder was 25.1 Kg/min and the AIPCS solution was sprayed at a rate of 1.6 Kg/min.

The AIPCS content was determined as before and was found to be the same as the theoretical value, within the experimental error, that is to say, there was no measurable loss of AIPCS during the processing.

Examples 2 and 3 demonstrate the advantage to be gained as regards reduced loss of AIPCS during processing by operating the process of the invention, in comparison with the process defined in Example 1.

Example 4

The detergent powders produced by the processes described in Examples 1, 2 and 3 were assessed for storage stability. They were sealed in non-laminated packs and stored at 30°C and 80% relative humidity for 18 weeks. Initially, and at 6 week intervals the content of AIPCS was analysed spectro-photometrically. The results are shown in Table II.

Table II

	Powder of Ex 1 (via slurry)		Powder of Ex 2 & 3 (via spraying)	
Storage Period (weeks)	AIPCS content	% loss	AIPCS content	% loss
0	0.0080	-	0.0062	-
6	0.0073	8.7	0.0058	6.8
12	0.0067	16.0	0.0057	7.5
18	0.0061	24.0	0.0057	7.5

It can be seen that the percentage loss of AIPCS is significantly lower when AIPCS is added to the powder by spraying, as in Examples 2 and 3 rather than by incorporating it in the crutcher slurry as in Example 1.

CLAIMS:

1. A process for the manufacture of a particulate detergent fabric washing product which comprises forming a spray-dried or granulated base powder and combining it with a photobleach compound, characterised in that the combination is effected by spraying a solution or suspension of the photobleach onto a powder comprising the base powder.

2. A process in accordance with claim 1 wherein the solution or suspension is sprayed onto the powder in a fluidised bed.

3. A process in accordance with claim 1 or claim 2 wherein the photobleach is a zinc or aluminium phthalocyanine sulphonate.

4. A process in accordance with any one of the preceding claims wherein the concentration of photobleach in the solution or suspension is from 0.001 to 0.2% by weight.

5. A process in accordance with any one of the preceding claims wherein the solution or suspension also contains an inorganic salt.

6. A process in accordance with claim 5 wherein the inorganic salt comprises sodium dihydrogen phosphate, sodium bicarbonate or sodium bisulphate.

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EUROPEAN SEARCH REPORT

0119746
Application number

EP 84 30 1006

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
D,A	EP-A-0 057 088 (PROCTER & GAMBLE LTD.) * Claims 1-4 *		C 11 D 3/00 C 11 D 3/39 C 11 D 3/395 C 11 D 17/00
A	FR-A-2 306 261 (PROCTER & GAMBLE CO.) * Page 5, line 30; claims 1, 2 *		

TECHNICAL FIELDS SEARCHED (Int. Cl. ³)

C 11 D 3/00
C 11 D 17/00

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	22-05-1984	SCHULTZE D

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
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